

CLAIMS

1. A method for continuous synthesis of a monoalkylhydrazine of formula

5 $\text{NH}_2\text{-NH-R (I)}$

in which R represents independently an alkenyl radical at C₂-C₆, an alkynyl radical at C₂-C₆, a linear alkyl radical at C₁-C₅ containing at least one imine function (-C=N-) or a linear or branched alkyl radical at C₁-C₆ carrying at least one
10 functional group selected from the group comprised of the radicals OH, alkoxy at C₁-C₆, C=NH, C≡N, phenoxy, COOH, COO-alkyl at C₁-C₆, phenyl or NR₃R₄, R₃ and R₄ each representing independently an alkyl radical at C₁-C₆ or forming a ring at C₂-C₆, wherein it comprises the following successive steps:

15 a) synthesizing the monoalkylhydrazine of formula I in a suitable reactor while causing to react in an alkaline medium and at a temperature in the range between 25 and 45 °C a monochloramine with an anhydrous amine of formula NH₂-R (II), R having the same significance as for formula I; then

20 b) demixing the solution obtained following step a in an organic phase and an aqueous phase by the addition of anhydrous sodium hydroxide under cooling so that the temperature of the demixing medium does not exceed the boiling points of the compounds; and

25 c) isolating from the organic phase thus obtained the monoalkylhydrazine of formula I.

30 2. A method according to claim 1, wherein, in step a, the formula II anhydrous amine/monochloramine molar ratio is in the range between 18 and 30.

3. A method according to either of claims 1 or 2, wherein the appropriate reactor to use in step a is a stirred tubular reactor.

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4. A method according to any of the preceding claims, wherein before step a the monochloramine is alkalized in a

mixer by the addition of a solution of sodium hydroxide in such a way that the weight concentration in sodium hydroxide is in the range between 2% and 6%.

5 5. A method according to claim 4, wherein the mixer is maintained at a temperature in the range between -10 and 5 °C.

10 6. A method according to any of the preceding claims, wherein the quantity of the anhydrous sodium hydroxide added during step b is such that the weight concentration in sodium hydroxide is in the range between 10% and 35%.

15 7. A method according to any of the preceding claims, wherein step c comprises the following successive steps:
i) isolating the unreacted anhydrous amine of formula II and a concentrated solution of the monoalkylhydrazine of formula I by distillation of the organic phase obtained following step b; then
20 ii) if necessary, purifying the aforesaid concentrated solution of the monoalkylhydrazine of formula I.

25 8. A method according to claim 7, wherein the aforesaid unreacted anhydrous amine of formula II recovered following step i is reinjected into the reactor of step a.

30 9. A method according to claim 7, wherein the concentrated monoalkylhydrazine solution of formula I is purified by distillation, said distillation being possibly preceded by a step of demixing into an organic phase and an aqueous phase by the addition of anhydrous sodium hydroxide in such a way that the weight concentration in sodium hydroxide is in the range between 30% and 50%.

35 10. A method according to any of the preceding claims, wherein the monochloramine is prepared according to a method comprised of the successive following steps:

α) preparing an aqueous sodium hypochlorite solution having a chlorometric degree in the range between 36° and 100°, possibly by the dilution of a hypochlorite solution having a chlorometric degree in the range between 100° and 120°; then

β) reacting a solution of ammonium hydroxide and of ammonium chloride with the aqueous sodium hypochlorite solution obtained following step α, in a slightly alkaline medium, at a temperature in the range between -15 and -7 °C, in order to form the aforesaid monochloramine.

11. A method according to claim 10, wherein the molar ratio of the ammonium hydroxide and ammonium chloride solution to the aqueous sodium hypochlorite solution advantageously lies between 2.5 and 3.

12. A method according to the claim 10 or 11, wherein the molar ratio of the ammonium chloride to the ammonium hydroxide advantageously lies between 0.1 and 1.75, advantageously it is approximately 0.65.